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<b>(21) International Application Number:</b> PCT/US92/04500 <b>(22) International Filing Date:</b> 28 May 1992 (28.05.92) <b>(30) Priority data:</b> 710,473      5 June 1991 (05.06.91)      US		<b>(81) Designated States:</b> AT (European patent), BE (European patent), CA, CH (European patent), CS, DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), HU, IT (European patent), KR, LU (European patent), MC (European patent), NL (European patent), PL, SE (European patent).
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<b>(54) Title:</b> DRY BLEACH COMPOSITION WITH IMPROVED DISPERSIBILITY  <b>(57) Abstract</b>  A dry, fabric bleaching composition, which leaves minimal residue in cold water washes, comprising (a) a bleaching-effective amount of a peroxygen bleach; (b) an alkaline builder material in an amount sufficient to provide an alkaline pH and building capacity; (c) about 0.05-0.5 % of a calcium stearate powder; and (d) 0 - about 5 % of a surfactant. A method of making the composition of the present invention is also disclosed, and comprises dry blending or agglomerating the peroxygen bleach and alkaline builder, as well as any other dry ingredients. To this is added the calcium stearate powder which is further blended until uniformly coated. Last, the liquids, especially nonaqueous liquids such as surfactant, are applied using a relatively coarse spray pattern, while continuing to mix, to result in a uniformly coated, free flowing dry mixture.		

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DRY BLEACH COMPOSITION  
WITH IMPROVED DISPERSIBILITY

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Background of the Invention

1. Field of the Invention

10        This invention relates to dry fabric bleaching products for household use, and more particularly to such dry bleach products based upon peroxygen bleaches, which are formulated to exhibit improved dispersibility/solubility in cold water.

15        2. Description of Related Art

      Bleaching compositions have long been used in households for the bleaching and cleaning of fabrics. Liquid bleaches based upon hypochlorite chemical species have been used extensively, as they are inexpensive, highly effective, easy to produce, and stable. However, the advent of modern synthetic dyes and the use of modern automatic laundering machines have introduced new requirements in bleaching techniques, and have created a need for other types of bleaching compositions. In order to satisfy this need, and to broaden and extend the utility of bleaches in household use, other bleach systems have been introduced in recent years, most notable are the peroxygen bleaches which generate hydroperoxide ion as the oxidizing species. A particularly preferred peroxygen bleach is sodium perborate which is suitable for a dry granular formulation. Preferably sodium perborate is combined with an alkalinity booster/builder such as sodium carbonate and is used as a laundry additive. Such a

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laundry additive composition is more fully described in US  
3,697,271 to Maddox, incorporated by reference herein.

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While these compositions have a demonstrated effectiveness,  
changes in laundering procedures often dictated by  
environmental concerns, can reduce their effectiveness. Cold  
water washes, for example, coupled with shortened wash cycles  
may hamper solubility/dispersion of sodium perborate/sodium  
carbonate formulations. Detergents or additives having high  
sodium carbonate levels readily form bridged hydrated product  
lumps when placed in piles and submerged in cold water. In a  
washing machine, these lumps can not be broken apart by  
agitation and leave residual product known as cold water  
residue (CWR). When the bulk solution temperature is low  
enough to cool the internal structure of the submerged pile  
below the melting points of the carbonate hydrates,  
precipitation of hydrates occurs in the saturated internal  
pile spaces and a bridged CWR mass is formed. CWR has been  
observed in the washer at temperatures as high as 75°F.  
Approximately 25% of all U.S. washloads are conducted at or  
below this temperature.

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Several references have addressed the question of improving  
cold water dissolution of laundry detergent compositions,  
however, these limit themselves to compositions containing  
detersive levels (above about 5%) of surfactant. Cala et al,  
US 4,196,095, describes and claims a dry blended,  
carbonate-based detergent comprising 30 to 90% of a builder  
salt, at least one third of which is sodium carbonate; sodium  
silicate; 5 to 30% of a surfactant, and 0.1 to 2% of magnesium  
stearate. The magnesium stearate is employed to reduce  
insoluble lumps formed when a detergent composition contacts  
cold water. Nakamura et al, US 4,970,017, claims a process

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5 for producing a granular detergent composition having high density and wherein one step of the claimed process comprises coating a disintegrated granular detergent composition with 0.5 to 5% by weight of a water-insoluble finely divided powder.

10 JP 60-96698 (Hara et al), describes a method for manufacturing a granular detergent composition wherein 0.5 to 5 weight percent of calcium stearate or other water insoluble powder is added to a granulated detergent product specifically to improve solubility in cold water. JP 62-228000 Saito et al, describes and claims a high density granular detergent  
15 composition, also employing a hydrophobic powder such as calcium stearate, as a means of improving cold water solubility. JP 64-20298 to Nakamura et al also describes and claims a high density granular detergent composition having as its object the attainment of a better cold water dissolution  
20 rate. This application points out one of the disadvantages in the use of hydrophobic fine powders to attain satisfactory dissolution rates, that is, while the hydrophobicity of the powder may aid dispersion, it can also impede dissolution.

25 Summary of the Invention

It is therefore an object of the invention to provide a dry peroxygen bleach formulation which exhibits minimal cold water residue and retains good flowability and pour qualities.

30 It is also an object of the invention to provide a dry peroxygen bleach formulation which is highly soluble in aqueous media at low temperature.

Other objects and advantages of the invention will become apparent from a review of the following description and the  
35 claims appended hereto.

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Briefly, the present invention is a dry, fabric bleaching composition comprising

- 5 (a) a bleaching-effective amount of a peroxygen bleach;
- (b) an alkaline builder material in an amount sufficient to provide an alkaline pH and building capacity;
- 10 (c) about 0.05-0.5% of a calcium stearate powder; and
- (d) 0 to about 5% of a surfactant.

The laundry additive composition of the present invention exhibits a dramatic reduction in cold water residue compared with similar compositions of the art having no calcium stearate powder. Surprisingly, the improvement in cold water residue does not result in a reduction in pour qualities or flowability of the composition, nor are the dissolution properties adversely affected.

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A method of making the composition of the present invention is also disclosed, and comprises dry blending the peroxygen bleach and alkaline builder, as well as any other dry ingredients. To this is added the calcium stearate powder which is further blended until completely dispersed, uniformly coating the other dry ingredients. Generally about 5-10 minutes in a tumble style mixer is required. Last, any liquids, particularly nonaqueous liquids such as surfactant, are applied using a coarse spray, while continuing to mix, to result in a uniformly coated dry mixture. Optimally, the initial dry blending step may be replaced by an agglomeration step wherein the oxidant and builder are coagglomerated with an agglomerating agent.

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If the agglomeration step option is utilized, it is preferred that the builder be preloaded with a low level (less than about 5%) surfactant prior to agglomerating with the oxidant.

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Description of the Drawings

5        Figures 1 and 2 are graphs showing the effect on bulk solution  
dissolution of various levels of calcium stearate powder (as  
calcium stearate). The data were obtained using a sodium  
perborate/sodium carbonate laundry additive matrix, and wash  
10       conditions were a water temperature of 4.5°C, 100 ppm hardness  
and five pounds of ballast. Results were obtained as  
conductance (in millisiemens/cm).

15       Fig. 1 is a graph showing the effect of 0.25 weight percent of  
calcium stearate on bulk solution dissolution compared to a  
control having no calcium stearate; and

20       Fig. 2 is a graph showing the effect of 0.5 weight percent of  
calcium stearate on bulk solution dissolution compared to the  
same control.

Detailed Description of the Invention

25       Briefly, the present invention is a dry, fabric bleaching  
composition comprising

- 30            (a) a bleaching-effective amount of a peroxygen  
bleach;
- (b) an alkaline builder material in an amount  
sufficient to provide an alkaline pH and building  
capacity;
- (c) about 0.05-0.5% of a calcium stearate powder; and
- (d) 0 to about 5% of a surfactant.

35       A first method of making the composition of the present  
invention is also disclosed, and comprises dry blending the  
peroxygen bleach and alkaline builder, as well as any other  
dry ingredients. To this is added the calcium stearate powder

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5 which is further blended until completely dispersed, uniformly  
coating the other dry ingredients. Generally about 5-10  
minutes in a tumble style mixer is required. Last, any  
liquids, particularly nonaqueous liquids such as surfactant,  
are applied using a coarse spray, which continuing to mix, to  
result in a uniformly coated dry mixture.

10 A second method of making the composition of the present  
invention is an agglomeration process wherein the oxidant  
material and alkaline builder are agglomerated prior to  
addition of the calcium stearate powder. In this method, the  
15 alkaline builder is first preloaded with surfactant, and any  
additional liquid additives. Thus surfactant is applied to  
the builder in a mixer, preferably, a tumble-style or falling  
curtain rotary mixer, and mixed sufficiently for the builder  
to substantially absorb the surfactant. Sufficient surfactant  
20 is applied to the builder to result in a final product (after  
agglomeration) surfactant content of 0 to about 5 wt. %, preferably about 0.1 - 3 wt.%. Generally on a surfactant to  
builder weight basis about 0-20 wt. % surfactant is applied to  
the builder, more preferably about 1-15 wt. %. The preloaded  
25 builder and oxidant are then charged to the agglomerator. Any  
agglomerating apparatus known to the art may be employed and  
preferred are rotary or vertical turbo agglomerators.  
Similarly, any agglomerating agent may be used, with sodium  
silicate and polyacrylates preferred. The agglomerate is then  
30 mixed with the calcium stearate powder and any additional dry  
ingredients in a mixing means, especially a rotary or tumble  
mixer.

35 In both processes, care must be taken to minimize physical  
contact between the calcium stearate and the surfactant, in  
order to ensure the efficacy of each component.



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Unless indicated to the contrary, all percentages, ratios, or parts are determined by weight.

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### Bleach

Preferred as bleaches are peroxygen or peracid bleaches in solid form. Preferred peroxygen bleaches include sodium percarbonate, sodium perborate, sodium phosphate peroxyhydrate, potassium permonosulfates and metal peroxides. Sodium perborate is most preferred and may be in the form of tetrahydrate or monohydrate. Bleach activators, also known as peracid precursors, can be included with the peroxygen compounds. Examples of activators include tetraacetyl ethylenediamine (TAED), nonanoyloxy benzene-sulfonate (NOBS), and nonanoylglycoylphenol sulfonate (NOGPS). NOGPS is disclosed, for example, in US patent 4,778,618 issued to Fong et al and in EP 373743 to Bolkan et al, the disclosures of which are incorporated herein by reference. If added, the peracid precursor is added in an amount effective to provide oxidizing power, and generally in a mole ratio to oxidant bleach of about 0.1:1 to 10:1. Peracid bleaches (including monoperacids and diperacids) may be advantageous in terms of bleaching performance. Examples include perazelaic and diperazelaic acids, diperoxydodecanedioic acid (DPBDA) and alkyl monoperoxysuccinic acid. Peracid bleaching species, and a method for their production, are described in U. S. patent 4,337,213 to Marynowski et al, the disclosure of which is incorporated herein by reference. The bleach is present in an amount sufficient to provide effective bleaching, e.g., from about 5 to 50% by weight active, preferably from about 8-25% by weight active, most preferably from about 10 to 13% by weight active depending on the bleaching species chosen.

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Alkaline Builder

5 An alkaline builder material is added to provide to a pH of  
between about 8-12. The builder also has the capacity to  
sequester or precipitate hardness ions (e.g.  $\text{Ca}^{2+}$  and  
10  $\text{Mg}^{2+}$ ). Alkali-metal carbonates, sesquicarbonates and  
bicarbonates are suitable builders, and preferred are sodium  
and/or potassium carbonates. The carbonate acts as the  
builder to remove divalent metal ions such as calcium, and  
15 additionally provides alkalinity and aids in soil removal.  
Generally, in terms of weight percent of the composition, at  
least about 25%, preferably 50%, most preferably 80% carbonate  
is employed. Higher levels can be employed, however, at  
20 levels greater than about 90% there is insufficient room for  
the other ingredients which contribute to the overall  
effectiveness of the composition.

20

Calcium Stearate Powder

Very low levels of a calcium stearate powder are important to  
significantly reduce the levels of CWR in the dry peroxygen  
bleaching matrix. Preferred are calcium stearates, available,  
25 for example, from The Synthetic Products Company (Synpro), of  
Cleveland, Ohio. Examples of particularly preferred calcium  
stearate are Synpro's finely-sized grades, especially grades  
15, 12B, 24-46, 114-36, NF and Food Grade. Preferably, the  
calcium stearate powder has a particle size distribution such  
30 that at least 95 percent is smaller than a US 200 mesh screen,  
and has a bulk density of about  $0.1\text{-}0.4\text{ g/cm}^3$ . More  
preferably the calcium stearate powder has a particle size  
distribution such that at least 99% is smaller than a US 200  
mesh screen, and 95% is smaller than a 400 mesh screen. A  
35 fine particle size is important to ensure effective coating of

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the other dry ingredients to result in a hydrophobic  
5 environment, thus mitigating cold water residue.

Granulometer measurements reveal that the most preferred  
calcium stearate fine powder has a median grain size of less  
than about 25 microns, preferably less than about 10 microns.

10

#### Liquid Additives

A surfactant may be provided to prevent "dusting" of the dry  
ingredients, particularly sodium carbonate, sodium perborate,  
15 and fluorescent whitening agents. It is most preferred to use  
at least one nonionic surfactant, especially C<sub>1-4</sub>  
alkoxylated aliphatic alcohols and C<sub>1-4</sub> alkoxylated alkyl  
phenols. Particularly preferred are ethoxylated/propoxylated  
C<sub>8-14</sub> alcohols. There should be at least about three alkoxy  
20 groups per alcohol, preferably at least about nine. Examples  
of preferred ethoxylated/propoxylated aliphatic alcohols are  
BASF Corporation's trademarked INDUSTROL, and PLURAFAC.  
Certain C<sub>1-4</sub> alkylene oxide copolymers such as ethylene  
oxide/propylene oxide copolymers are also preferred as  
25 surfactants. These are exemplified by BASF's trademarked  
PLURONIC series. Other suitable nonionic surfactants are  
polyethoxylated alcohols manufactured and marketed by the  
Shell Chemical Company under the trademark "NEODOL". Examples  
of preferred NEODOLS are NEODOL 25-7 which is a mixture of 12  
30 to 15 carbon chain length alcohols with about 7 ethylene oxide  
groups per molecule, NEODOL 23-65, a C<sub>12-13</sub> mixture with  
about 6.5 moles of ethylene oxide, and NEODOL 25-9, a C<sub>12-15</sub>  
mixture with about 9 moles of ethylene oxide. Also useful are  
a trimethyl nonyl polyethylene glycol ether, manufactured and  
35 marketed by Union Carbide Corporation under the Trademark  
TERGITOL TMN-6, and an octyl phenoxy polyethoxy ethanol sold  
by Rohm and Haas under the Trademark TRITON X-114. Total

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5 surfactant content may range from 0 to about 5%, preferably  
from about 0.1 to 3%, more preferably from 0.2 to 1% and most  
preferably from about 0.2 to 0.3%. It is to be noted that  
higher levels of surfactant, and/or its application as an  
overly fine spray, tends to cause agglomeration of the calcium  
stearate powder, thus severely reducing its effectiveness. It  
10 is also preferred to a ratio of surfactant to calcium stearate  
powder be from about 3:1 to 1:5.

#### Adjuncts

15 Adjuncts may be added in an amount of from 0 to about 5% and  
are useful to improve or enhance efficacy, aesthetics and/or  
consumer acceptance of the overall formulation. Enzymes are a  
particularly preferred adjunct, and may be selected from  
amylases, proteases, cellulases, and lipases. The hydrolytic  
enzyme should be present in an amount of about 0.01-2%, more  
20 preferably about 0.5-1%, by weight of the detergent. Mixtures  
of any of the foregoing hydrolases are desirable, especially  
protease/amylase blends.

25 Dyes, such as Monastral blue and anthraquinone dyes (such as  
those described in Zielske, U.S. 4,661,293, and  
U.S. 4,746,461), and pigments, e.g. titanium dioxide and  
ultramarine blue which are also suitable colorants, can be  
selected. Anti-redeposition agents, such as  
carboxymethylcellulose, are potentially desirable.  
30 Sequestrants, such as EDTA, citric acid, polyphosphonates,  
aminopolyphosphonates, and the like, may also be desirable to  
complex transition metal ions which can destabilize bleaches.

Fluorescent whitening agents (FWAs) are desirable  
components for inclusion in bleaching formulations, as they  
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counteract the yellowing of cotton and synthetic fibers. FWAs are absorbed on fabrics during the washing and/or bleaching process. FWAs function by absorbing ultraviolet light, which is then emitted as visible light, generally in the blue wavelength ranges. The resultant light emission yields a brightening and whitening effect, which counteracts yellowing or dulling of the bleached fabric. Such FWAs are available commercially from sources such as Ciba Geigy Corp. of Basel, Switzerland, under the trade name "Tinopal". Incorporation of the FWAs may be afforded by mixing a binding agent and bulking agents e.g.,  $\text{Na}_2\text{SO}_4$ , and colorants. The mixture is then compacted to form particles, which are admixed into the bleach product. If added, the FWA particles may comprise from about 0.1% to 1% by weight of the composition.

A fragrance which imparts a pleasant odor to the bleaching composition is generally included. As fragrances are subject to oxidation by bleaches, they may be protected by encapsulation in polymeric materials such as polyvinyl alcohol, or by absorbing them into starch or sugar and forming them into beads. These fragrance beads are soluble in water, so that fragrance is released when the bleach composition is dissolved in water, but the fragrance is protected from oxidation by the bleach during storage.

Buffering, building, and/or bulking agents may also be present. Boric acid and/or sodium borate are preferred agents to buffer the pH of the composition. Other buffering agents and cobuilders such as sodium and potassium silicate, sodium phosphate, sodium tripolyphosphate, sodium tetrakisphosphate, aluminosilicates (zeolites), and organic builders such as sodium sulfosuccinate may be added. Optionally, fillers such as sodium sulfate are added. Buffer, builder, and bulking agents are included in the product in particulate form such

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that the entire composition forms a free-flowing dry product.  
5 Buffers and cobuilders and/or bulking agents may range from 0  
to about 80%, preferably 10-50% by the weight of composition.

For a dry-blend process, liquid ingredients, including  
liquid adjuncts, are preferably sprayed onto the dry  
ingredients after application of the calcium stearate powder,  
10 and more preferably, as a final step. It is important in the  
application of liquids, particularly nonaqueous liquids such  
as nonionic surfactants, that the application be carried out  
in such a way as to prevent agglomeration of the calcium  
stearate. For the preferred method of applying liquids last,  
15 the liquid application apparatus should be selected to deliver  
a relatively coarse spray, and mixing should be gentle to  
moderate. A desired coarse spray may be obtained by a pump-fed  
non-atomizing nozzle, having a fan-shaped spray pattern. An  
example is a nozzle sold under the name T-Jet 11002. It is  
20 within the scope of the dry-blending-process of the present  
invention, however, to preload surfactant or other liquids  
onto sodium carbonate prior to blending in the remaining dry  
ingredients, and prior to adding any remaining liquid  
ingredients. If preloading is undertaken, the spray density  
25 of surfactant is less important as long as the carbonate is  
sufficiently mixed during and after spraying to assure maximum  
absorption of surfactant. It is preferred to employ a  
tumble-style or falling curtain rotary mixer for the  
preloading, and the surfactant may be applied using an air-fed  
30 atomizing spray apparatus.

Dry adjuncts may be added at any time during the process,  
for example with the sodium perborate and carbonate or after  
the addition of all liquid ingredients.

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## FORMULATION EXAMPLES

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<u>Ingredient</u>	<u>Weight Percentage</u>
Na <sub>2</sub> CO <sub>3</sub>	60-90
Sodium Perborate	2-30
Calcium Stearate	0.05-0.5
10 Surfactant	0-5
Enzyme	0-2.0
Brightener	0-0.5
UMB	0-0.2
Fragrance	0-0.3

15

Experimental

Table I below shows the importance of particle size distribution of the calcium stearate powder on CWR. The measurement of CWR is accomplished by placing a measured quantity of laundry composition, as a single pile, in the bottom of a washing machine. Ballast (10 lbs. of polycotton pillow cases) is piled on top of the composition. The washing machine is set to run a gentle eight minute cycle with 4.5°C incoming wash water. At the end of a complete wash cycle the ballast is removed and undissolved composition collected and weighed.

25

The data of Table I was obtained by placing 110 g of a sodium perborate/sodium carbonate - based additive formulation, containing 0.25 weight percent calcium stearate as the calcium stearate powder, in a pile in a washing machine. Ten pounds of ballast, in the form of polycotton pillowcases were added, and the wash was conducted at a water temperature of 4.5°C, with the results shown in the table. All mesh sizes are US mesh.

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Table I

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	<u>Grade</u>	<u>Hydrophobic Powder</u>	<u>CWR (g)</u>
		<u>Size Distribution</u>	
	A	60% through 40	83.3
	B	80% through 200	35.4
	C	99% through 200	0.0
10	D	95% through 200	0.1
	E	99% through 200	2.3
	F	99% through 200	1.7
	Control (1)		86.4

(1) No calcium stearate

15

The results show that grades A and B were insufficiently finely sized to achieve an acceptable reduction in CWR. Grades C-F, however, yielded an acceptable level of CWR. The results of Table I are accurate to within 5.0 g, thus C-F should be considered equal to each other. Generally, less than about 30 g, preferably less than about 10 g and most preferably less than about 5 g is acceptable. Alternatively, CWR is expressed as a percentage by comparing final weight of laundry product with the initial weight. Expressed as a percentage of additive remaining after a wash cycle, less than about 30% more preferably, less than about 10% and most preferably less than about 5% CWR is acceptable.

20

Table II shows the minimum amount of calcium stearate powder (as calcium stearate) necessary to achieve acceptable CWR levels. The calcium stearate was added to a sodium carbonate/sodium perborate formulation as in the Example, and had a size distribution of 99% through a 200 mesh screen. Experimental conditions were as given for Table I above, except more vigorous agitation was employed in the wash cycle.

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Table II shows that for a sodium carbonate/perborate composition, at least about 0.05 wt. %, preferably at least about 0.15 wt. % calcium stearate is important to reduce CWR to acceptable levels.

Table II

<u>Wt. % Calcium Stearate</u>	<u>CWR (g)</u>
0.0	78.6
0.025	74.4
0.050	28.6
0.150	0.3
0.250	0.2
0.500	0.1

For perborate/carbonate laundry additive formulations, a level of calcium stearate powder above 0.5 wt %, the product bridges or clumps slightly as it is poured with the degree of clumping becoming worse with increasing calcium stearate powder level. This phenomenon is seen in the results of Table III which presents angle of repose data for compacted and uncompact sodium carbonate/perborate samples containing calcium stearate, poured in a tilt plate apparatus. The tilt plate is a twelve inch long smooth surfaced-plate, upon which is placed about 100 g of sample, in a single pile. The plate is tilted to various angles and the angle at which the sample begins to flow is noted.

At a 0.25 weight percent calcium stearate level, the pourability of the formulation does not change. The 0.5 weight percent level produces a slight change relative to a stearate-free sample, thus may be considered a maximum level with respect to product pour/flow characteristics.

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Table III

5	<u>Angle of Repose</u>		
	<u>Weight % Calcium Stearate</u>	<u>Uncompacted Sample</u>	<u>Compacted Sample</u>
10	0.00	20°	21°
	0.25	20°	21°
	0.50	22°	22°
	1.00	24°	26°
	3.00	25°	28°
	5.00	25°	28°
	10.00	26°	30°

15 Once dispersed from the bottom of the washer by the agitator the individual granules of additive composition must dissolve in the bulk solution. Owing to the hydrophobic nature of the calcium stearate powder, an excess (above about 0.5%) can interfere with bulk solution dissolution of the additive, as the additive particles become sufficiently coated to prevent them from dissolving. Japanese patent application 62-22800 teaches that while stearate alone is effective for reducing CWR, it also inhibits bulk solution dissolution.

25 Figures 1 and 2 illustrate bulk solution conductivity profiles for a mixture of sodium carbonate/sodium perborate and calcium stearate at 4.5°C. Bulk solution dissolution is measured by first filling a washing machine with water at the desired temperature. A 100 g sample was added to the water in the washer tub, and conductance was measured using a Radiometer America Conductivity Meter, Model CDM-83. Conductance is expressed in millisiemens/cm, and increases with dissolution of the sample.

35 All materials were added to bulk solution (5 lbs ballast, 100 ppm hardness). At the 0.25 weight percent calcium stearate level, the bulk solution dissolution is equivalent to the

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5       stearate-free control after eight minutes (both were 84%  
dissolved). A reduction in solution conductivity is seen for  
the 0.5 wt % stearate level. Based on a conductance vs.  
sodium carbonate level calibration curve (not shown) the pure  
sodium carbonate is 84% dissolved after eight minutes while  
10       the sodium carbonate with 0.5 wt. % stearate is 79%  
dissolved. Therefore, the 0.5 wt. % calcium stearate  
prevents approximately 5 grams (5%) of sodium carbonate from  
dissolving after eight minutes. The 0.5% level thus is a  
maximal level (for a sodium perborate/sodium carbonate  
additive formulation) above which the beneficial effects of  
15       the stearate on additive dissolution is reduced.

#### Cleaning Performance

20       To verify that calcium stearate does not reduce performance  
when a dry bleach composition (containing sodium perborate)  
is added directly to solution, standard condition (20, 35,  
and 50°C; 100 ppm hardness) performance and multi-cycle  
whitening studies were conducted. A leading  
25       commercially-available phosphate detergent served as the  
detergent matrix, and a leading commercially-available sodium  
perborate/sodium carbonate laundry additive, to which was  
added 0.0 and 0.5 weight percent calcium stearate, was  
tested. The treatments were added directly to solution to  
prevent lumping. Soil removal, whitening and redeposition  
30       were each measured colorimetrically by comparing reflectance  
measurements on swatches of fabric before and after washing.  
Whitening was measured after one, three and five cycles while  
redeposition was measured once after five cycles. Soil  
removal was measured after one cycle. As seen in the stain  
35       and soil averages presented in Table IV, the calcium stearate  
does not reduce performance. All results are shown for the  
average of the three wash temperatures. No reduction was

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5 found on any of the individual performance attributes as well  
(such as grass stains on cotton fabric or clay soil on  
polycotton fabric).

Table IV

	% Soil Removal			Sebum/ 3-Fabric <sup>(3)</sup>
	<u>3-stain</u> <sup>(1)</sup>	<u>2-soil</u> <sup>(2)</sup>		
Additive	75.7	81.9		86.0
Additive + 0.5 wt. % stearate	76.7	83.6		87.7
15 LSD, 95% t-test	1.7	3.4		1.4
	Whitening			Redeposition
	<u>Cycle 1</u>	<u>Cycle 3</u>	<u>Cycle 5</u>	<u>Cycle 5</u>
Additive	17.7	19.5	21.0	-2.9
Additive + 0.5 wt. % stearate	17.2	18.8	20.8	-3.1
20 LSD, 95% t-test	0.8	1.1	0.6	1.4

(1) three different proteinaceous stains

(2) two particulate soils

(3) cotton, polycotton and polyester

25 Table V lists the laundry performance improvements achieved  
when the additive containing 0.5 wt. % calcium stearate is  
placed at the bottom of a washer for a cold water wash.  
Because the control formula lumps, it provides less  
alkalinity and brighteners to the wash resulting in the  
30 significantly reduced sebum and whitening performance.  
Given the CWR results for the 0.15 wt. % and 0.25 wt. %  
stearate levels presented in Table II, it is expected that  
the Table III results would be achieved by the additive  
plus 0.25 wt. % stearate formula as well.

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Table V

	<u>Wash Condition</u>	<u>Treatment</u>	<u>%SR (E)</u>
5	Sebum/cotton	with stearate	72.6
	21°C, regular	without stearate	67.9
	agitation	LSD, 95% t-test	2.5
	Sebum/polyester	with stearate	95.9
	21°C, regular	without stearate	91.8
10	agitation	LSD, 95% t-test	3.0
	Sebum/3-fabric	with stearate	82.8
	average	without stearate	79.6
	21°C, regular	LSD, 95% t-test	1.4
	agitation		
15	Whitening	with stearate	10.7
	12.8°C, regular	without stearate	9.1
	agitation	LSD, 95% t-test	1.0
	Whitening	with stearate	8.9
	4.5°C, regular	without stearate	6.8
	agitation	LSD, 95% t-test	0.8

20 Tables VI and VII illustrates the importance of the method of  
the present invention. Mixing time of dry ingredients and  
order of addition of surfactant and/or any liquids (e.g.,  
fragrance) can impact stearate dispersion, thereby affecting  
CWR. The product of Table VI was made as a 10 lb.  
25 dry-blended batch in a tumble mixer.

Table VI

	<u>Sample</u>	<u>Mixing Time (Min)</u>	<u>CWR(g)</u>
30	A	5	51.5
	A	10	4.8
	B	5	52.9
	B	10	50.8
	B	15	23.0

35 A = perborate plus 0.25 wt. % calcium stearate  
B = A plus 0.40 wt. % nonionic surfactant/fragrance

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5 Table VI shows that a minimum of ten minutes of mixing (in a small tumble mixer with gentle-moderate mixing) the dry ingredients is necessary to attain an acceptable CWR value. Table VI also illustrates the disadvantages of using a fine surfactant spray, as this tends to cause agglomeration of the calcium stearate particles and increase CWR unless mixing is continued for a long time. The spray apparatus used to  
10 obtain the mixture of Table VI was an atomizing sprayer embodying a pressure of about 25 psi at 2 g/min delivery. These spray conditions are accordingly not preferred where surfactant is added last.

15 A scale up was run in a 100 lb. batch Pelligrini mixer (a baffled, tumble mixer) set to provide vigorous mixing. A surfactant/fragrance mixture was applied as a coarse spray using a T-Jet 11002 Nozzle at 40 psi and 300 g/min delivery. A substantial decrease in CWR was evidenced by these process  
20 parameters, as shown in Table VII.

Table VII

	Sample	<u>Mixing Time (Min)</u>	CWR(g)
25	A	5	0.49
	A	10	0.45
	B	5	3.51
	B	10	1.05
	B	15	1.41

30 A = perborate plus 0.25 wt. % calcium stearate  
B = A plus 0.40 wt. % nonionic surfactant/fragrance

Both Tables VI and VII show that dispersion of stearate into the dry ingredients is important in achieving good CWR. The five minute value of Sample A of Table VII is sharply  
35 improved owing to the more rapid dispersion of stearate in the larger, more vigorous Pelligrini mixing apparatus. The

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5 coarser spray also improved CWR of the B samples, apparently  
by sharply reducing the tendency of the fine powder to  
agglomerate. Generally, only five minutes of mixing the dry  
ingredients, followed by another five minutes after  
surfactant addition, is needed to achieve acceptable CWR  
values.

10 While described in terms of the presently preferred  
embodiments, it is to be understood that such disclosure is  
not to be interpreted as limiting. Various modifications and  
alterations will no doubt occur to one skilled in the art  
after having read the above disclosure. Accordingly, it is  
15 intended that the appended claims be interpreted as covering  
all such modifications and alterations as fall within the  
true spirit and scope of the invention.

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Claims

1. A dry bleaching composition comprising
  - (a) a bleaching-effective amount of an oxidant, selected from the group consisting of peroxygen and peracid bleaches, and mixtures thereof;
  - 5 (b) a building-effective and alkalinity-adjusting amount of an alkaline builder;
  - (c) 0.05 to 0.5 weight percent of a calcium stearate powder, having a particle size distribution such that about 95% is smaller than US 200 mesh; and
  - 10 (d) 0 to about 5 percent surfactant.
2. The composition of claim 1 characterized by a reduction in conductance of no more than about 5%, and a cold water residue of no more than about 30%.
3. The composition of claim 1 wherein
  - the oxidant is sodium perborate, and the alkaline builder is sodium carbonate.
4. The composition of claim 3 wherein
  - the sodium carbonate is present in an amount of about 25-80 percent, and the sodium perborate is present in an amount of about 5-25%.
5. The composition of claim 1 and further including
  - about 0.1 to 5% adjuncts, selected from the group consisting of enzymes, coloring agents, brighteners, fragrances, buffering agents, fillers, sequestrants and
  - 5 mixtures thereof.



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6. The composition of claim 5 wherein  
said enzyme is selected from the group consisting of  
proteases, amylases, lipases, cellulases, and mixtures  
thereof.

7. The composition of claim 1 wherein  
the surfactant is a nonionic surfactant selected from  
the group consisting of ethoxylated alcohols, alkylene oxide  
polymers and mixtures thereof.

8. The composition of claim 1 and further including  
about 10-50 % cobuilder.

9. The composition of claim 1 and further including  
an effective amount of a peracid precursor.

10. A laundry additive particle comprising

(a) a core material consisting of an oxidant, an alkaline  
builder, or a mixture thereof;

5 (b) a plurality of particles of calcium stearate, having  
a particle size distribution such that about 95% are smaller  
than US 200 mesh, adsorbed onto the surface of said core; and

(c) 0 to about 5% surfactant absorbed onto the surface of  
said core.

11. The particle of claim 10 wherein  
the oxidant is sodium perborate, and the builder is  
sodium carbonate.

12. The composition of claim 11 wherein  
the sodium carbonate is present in an amount of about  
25-80 percent, and the sodium perborate is present in an  
amount of about 5-25%.

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13. The particle of claim 10 wherein

the surfactant is a nonionic surfactant selected from the group consisting of ethoxylated alcohols, alkylene oxide polymers and mixtures thereof.

14. A process for making a dry oxidant bleaching composition in steps comprising

(a) dry blending, in a mixing means, dry ingredients comprising about 5 - 25% oxidant, about 25-80% builder, and 0 to about 5% adjuncts;

(b) adding about 0.05 to 0.5% calcium stearate, having a particle size distribution such that about 95% is smaller than US 200 mesh, while continuing to blend until the dry ingredients are uniformly coated with calcium stearate; and

(c) applying to the dry ingredients a liquid ingredient selected from 0 to about 5% liquid surfactants, 0 to about 5% liquid adjuncts, and mixtures thereof, with continued blending until a uniform composition is attained.

15. The process of claim 14 wherein

the liquid surfactant is applied using a coarse spray with a non-atomizing spray apparatus.

16. A process for making a dry oxidant bleaching composition comprising

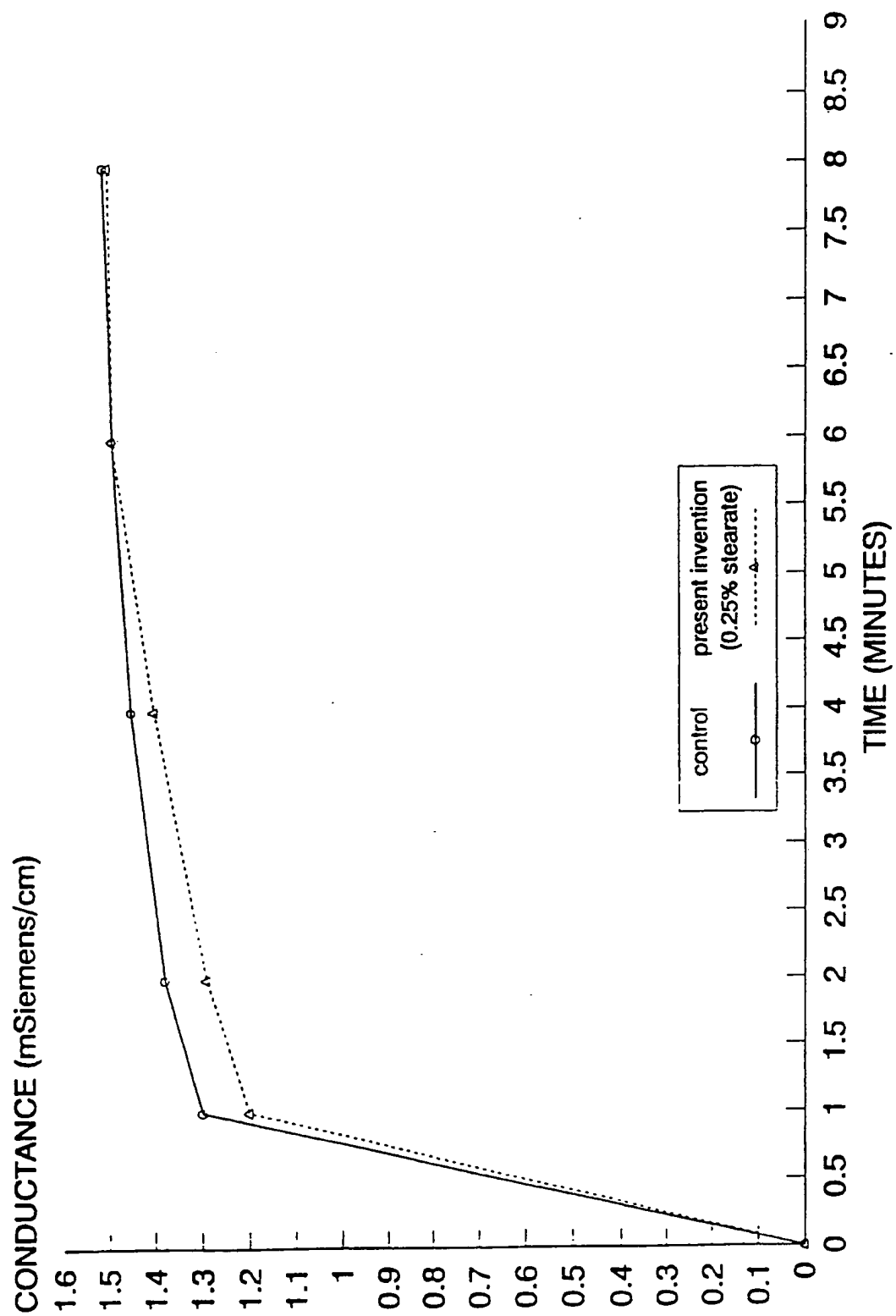
(a) preloading a quantity of liquid surfactant onto a quantity of an alkaline builder to result in 0 to about 20% surfactant being present on a weight basis relative to builder;

(b) coagglomerating a quantity of an oxidant bleach and said quantity of preloaded alkaline builder with an agglomerating agent;

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(c) applying 0 to about 1% of liquid adjuncts to the coagglomerate of (b), the liquid adjuncts being applied as a coarse spray; and

5 (d) blending the resulting agglomerate of (c) with about 0.05 to 0.5% of a calcium stearate powder having a particle size distribution such that about 95% is smaller than US 200 mesh, and continuing the blending until the calcium stearate has effectively coated the agglomerate.

**Fig. 1**

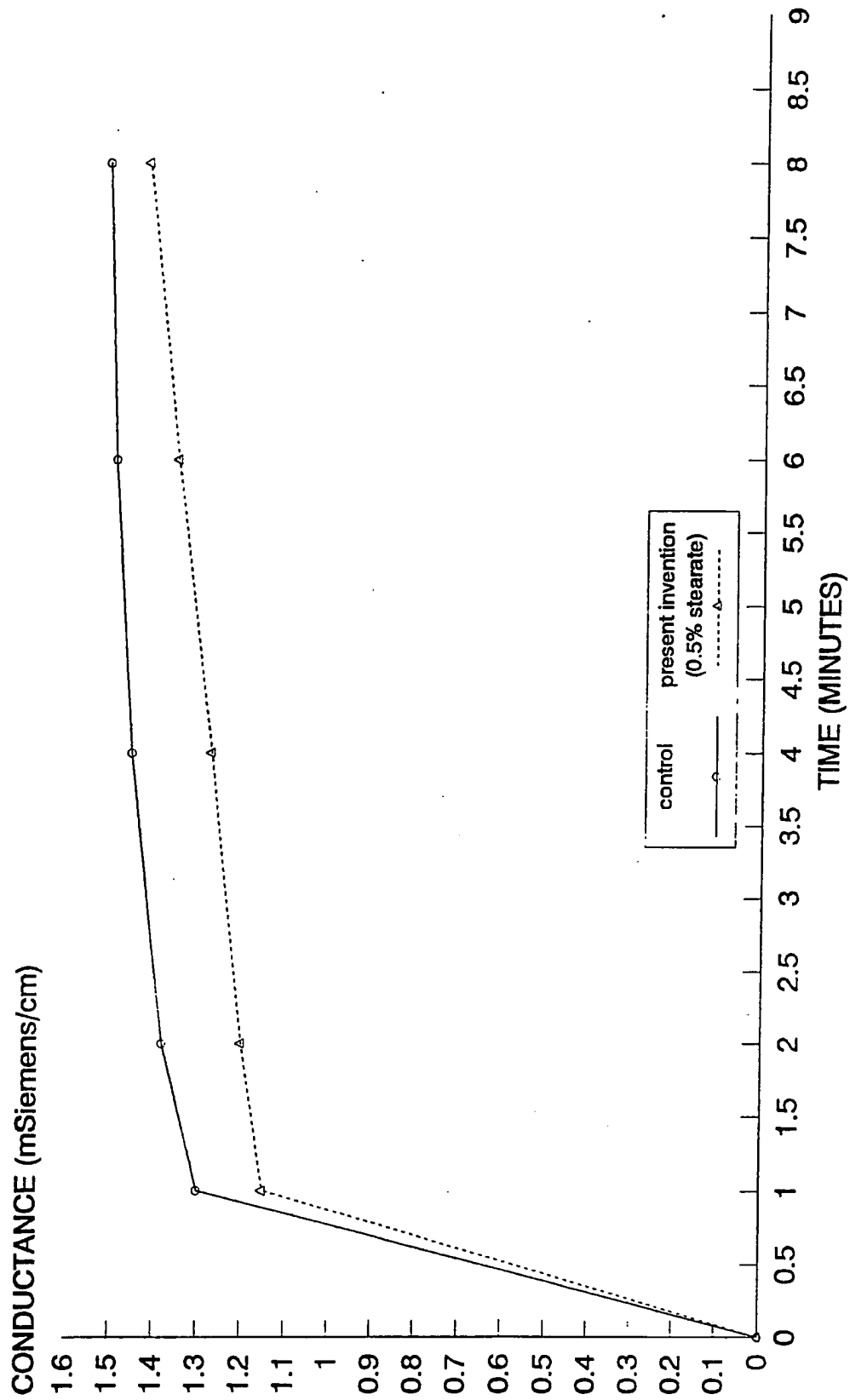


Fig. 2

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